

**SYNTHESIS AND CHARACTERIZATION OF SILICON CARBIDE FROM
COCONUT SHELL (*Cocos nucifera*) AND ITS POTENTIAL IN
PHOTOVOLTAIC CELL APPLICATION**

IAN JASPER R. AMPELOQUIO

An Undergraduate Thesis Submitted to the Faculty of the Department of Chemistry,
College of Arts and Sciences, Central Luzon State University,
Science City of Muñoz, Nueva Ecija, Philippines
in Partial Fulfillment of the Requirements
for the Degree of

BACHELOR OF SCIENCE IN CHEMISTRY

JUNE 2018

ACCEPTANCE SHEET

The undergraduate thesis entitled **SYNTHESIS AND CHARACTERIZATION OF SILICON CARBIDE FROM COCONUT SHELL (*Cocos nucifera*) AND ITS POTENTIAL IN PHOTOVOLTAIC CELL APPLICATION** prepared and submitted by **IAN JASPER R. AMPELOQUIO** in partial fulfillment of the requirements for the degree of **BACHELOR OF SCIENCE IN CHEMISTRY**, is hereby accepted.


NELSON M. PANAJON
Adviser

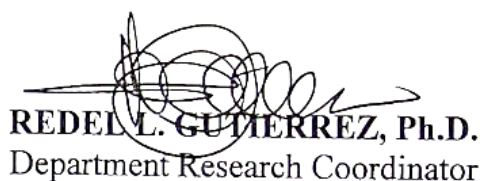
06/11/2018

Date Signed


NERISSA G. RAMOS, M.Sc.
Critic

06/18/2018

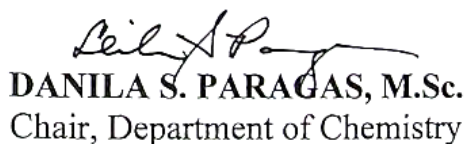
Date Signed


REIDEL L. GUTIERREZ, Ph.D.
Department Research Coordinator

06/18/2018

Date Signed

Accepted as partial fulfillment of the requirements for the degree of **BACHELOR OF SCIENCE IN CHEMISTRY**


DANILA S. PARAGAS, M.Sc.
Chair, Department of Chemistry

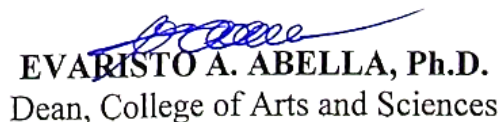
6/18/2018

Date Signed


RICH MILTON R. DULAY, M.Sc.
College Research Coordinator

06/18/2018

Date Signed


EVARISTO A. ABELLA, Ph.D.
Dean, College of Arts and Sciences

06/18/2018

Date Signed

BIOGRAPHICAL SKETCH

Ian Jasper Ramones Ampeloquio, the author of this paper and the researcher of this study, is a native of Dagupan, San Mateo, Isabela. Born on October 07, 1997, he finished his primary education at the Dagupan Elementary School as the class valedictorian on March 2010. He took his secondary education at the Eveland Christian College of San Mateo, Isabela and graduated on March 2014. As of writing, he studies at Central Luzon State University, Nueva Ecija taking up Bachelor of Science in Chemistry.

He is a recipient of CHED-SUC's Scholarship for the first and second semesters of academic school year 2017 – 2018. He was in the college baseball team for the first semester of academic school year 2016 – 2018.

ACKNOWLEDGEMENT

Bachelor of Science in Chemistry was really one of the most rigorous in its field as being proven by my four-year journey throughout this course. It will be even tougher without the help of all the people that made this point possible.

It is my utmost pleasure to thank the people who made this study possible. To my adviser, Sir Nelson M. Panajon for assisting in overnight experiments and being a forbearing, considerate, supportive, and dependable adviser that guided me to let me handle all the pressure of my last semester just fine. To Ma'am Nerissa C. Ramos for being a kind, understanding and supportive critic. To Dr. Joel R. Salazar for being a very supportive, resolute, and understanding professor that lead me to be part of this graduating class of 2017-2018 despite being initially delayed by one of subjects. To Dr. Juvy J. Monserate for his guidance and letting us use his laboratory providing sophisticated and even the newest equipment available that are needed in this study. I also want to thank Prof. Danila S. Paragas for allowing and assisting us in doing overnight experiments during the early stages of the study; and to Dr. Redel L. Gutierrez for his patience as our Department Research Coordinator.

I want to thank my grandparents for making my college life possible, providing most of the financial support and for all the things they did to support me in pursuing this course. To my parents, my sister, and to Ms. Florida D. Calaranan for providing emotional support and for believing in me that made it possible for me to stay positive regardless of the problems that I've encounter in conducting the study.

To my very supportive classmates and lab mates: Andrea Nicole P. Agustin, Arwil Nathaniel R. Alfonso, Lorie Ann Anda, Irish A. Bayudan, Aurelio L. Cardozo Jr.,

Loidel E. Gagelonia, Sahara Krisha T. Labiaga, Nathaniel Glenn C. Martin, Erica Jhoie Rubrico, Paul Michael A. Sanchez, and Arvin Luis M. Santos for all the support and help in the academics and my study. Especially for Paul, Au, and Arwil for spending all the sleepless laboratory nights and accompanying me in my extra laboratory hours. To Mr. Brilliant King R. Vergara for helping me in preparing and giving me advises in my thesis defense. Also, to Ms. Marilou M. Sarong for joining us in our overnight experiments and for teaching and assisting us in the statistical analysis that plays a vital role in this study.

And above all, I would like to thank the Almighty God for giving me all the blessings, for guiding me away from danger, and for answering all the prayers in the right time.

TABLE OF CONTENTS

	Page
TITLE PAGE	i
ACCEPTANCE SHEET	ii
BIOGRAPHICAL SKETCH	iii
ACKNOWLEDGEMENT	iv
LIST OF TABLES	viii
LIST OF FIGURES	ix
LIST OF APPENDICES	x
LIST OF APPENDIX FIGURES	xi
ABSTRACT	xii
INTRODUCTION	1
Background of the Study	1
Significance of the Study	2
Objectives of the Study	3
Time and Place of the Study	3
Scope and Delimitations of the Study	4
REVIEW OF RELATED LITERATURE	5
Coconut Shell	5
Silicon Carbide	9
Synthesis of SiC	13
Application of Silicon Carbide	15
Magnesiothermic Reduction Method	16
Photovoltaic Cell (Solar cell)	18
METHODOLOGY	21
Materials and Reagents	21
Experimental Procedure	21
Carbonization of Coconut shell	22
Synthesis of SiC from Carbonized Coconut Shell	22

Characterization and Solar Cell Potential of Products	23
Doping of Produced SiC	23
RESULTS AND DISCUSSION	24
Properties of Synthesized SiC	24
SUMMARY CONCLUSION, AND RECOMMENDATIONS	35
Summary	35
Conclusion	36
Recommendations	37
LITERATURE CITED	38
APPENDICES	44

LIST OF TABLES

TABLE		Page
1	Elemental Analysis of coconut shell	6
2	Chemical Composition of CSA after Combustion	7
3	Comparison of Several Important Semiconductor Material Properties	10
4	Different Treatments Involving the Synthesis of SiC via MR method	22
5	Conductance and Band Gap Energy	26
6	Conductance and Band Gap Energy of Phosphorus Doped SiC	27
7	Major IR peaks of the different treatments	31

LIST OF FIGURES

FIGURE		Page
1	Coconut shells bought from local market of the Science City of Muñoz, Nueva Ecija	5
2	FTIR of Carbonized Coconut Shell	8
3	Silicon Carbide (laboratory grown) from Ontario, Canada	9
4	Structure of 3C-SiC (right) and SEM micrograph of zinc-blende crystal structure	11
5	FTIR spectra for a 1.1 μm thick 3C-SiC film	12
6	FTIR Spectra of (a) SiC NP, (b) SiC – 500 NP, annealed at 500 ⁰ C, (c) SiC – SiO ₂ annealed at 700 ⁰ C, and (d) SiC-SiO ₂ -NH ₂ NP.	13
7	Solar energy conversion process	19
8	Different form of coconut shell: (a) dried coconut shell, (b) crushed coconut shell, (c) carbonized coconut shell	24
9	Synthesized SiC with different treatments (a) control, (b) T1, (c) T2, (d) T3, and (e) T4	25
10	FTIR Spectra of control	28
11	FTIR Spectra of Treatment 1	29
12	FTIR Spectra of Treatment 2	29
13	FTIR Spectra of Treatment 3	30
14	FTIR Spectra of Treatment 4	30
15	SEM micrographs of 1:3 SiC with different magnifications (a) 200 μm , (b) 100 μm , and (c) 50 μm	32
16	EDX spectra of 1:3 SiC with the elemental composition reported in percent mass.	33

LIST OF APPENDICES

APPENDIX NO.		Page
1	Documentation	45
2	Uv-Vis spectroscopy measurements	48
3	Band gap energy and recovery calculations	40
4	Statistical analysis of the band gap energies and conductance	57

LIST OF APPENDIX FIGURES

APPENDIX FIGURE		Page
1	Production of carbonized coconut shell	45
2	Carbonized coconut shell (before grinding)	45
3	CCS after grinding	45
4	CCS boiling in HCl	45
5	Synthesis of SiC of different treatments	46
6	Synthesis of SiC before HCl and HNO ₃ washing	46
7	Synthesized SiC (1:1) after calcination	46
8	KBr pelletized for FTIR analysis	46
9	Thermo Scientific Nicolet 6700 FTIR used in FTIR analysis of the products	47
10	Horiba SZ-100 used in conductance measurement of the samples	47
11	UV-Vis measurement of synthesized SiC of the different treatments (a) control, (b) T1, (c) T2, (d) T3, (e) T3.	48
12	UV-Vis Measurement of 0.2 M Na ₂ HPO ₄ Doped T4	48
13	UV-Vis Measurement of 1.5 M Na ₂ HPO ₄ Doped T4	49
14	UV-Vis Measurement of 2.0 M Na ₂ HPO ₄ Doped T4	49
15	Statistical analysis of the band gap energy (left) and the conductance (right) of the different SiC treatments.	57
16	Statistical analysis of the band gap energy (left) and the conductance (right) the treatment 4 doped with different concentrations of Na ₂ HPO ₄	58

ABSTRACT

IAN JASPER R. AMPELOQUIO., Department of Chemistry, College of Arts and Sciences, Central Luzon State University, Science City of Muñoz, Nueva Ecija, Philippines. **JUNE 2018, SYNTHESIS AND CHARACTERIZATION OF SILICON CARBIDE FROM COCONUT SHELL (*cocos nucifera*) AND ITS POTENTIAL IN PHOTOVOLTAIC CELL APPLICATION**

Adviser: NELSON M. PANAJON

Background: Coconut industry is one of the dominant sectors in Philippine agriculture. Being one of the largest industries, coconut processing often leaves large amount of agricultural wastes. Coconut shells contains relatively-high amount of silica and carbon, sources which are important in the synthesis of Silicon Carbide (SiC) as a valuable material that can be used in different industries. The synthesized Silicon Carbide was tested for its potential in solar photovoltaic cell application by measuring the band gap energy. A semiconductor for solar photovoltaic cell must have a band energy gap in the range of 1.1 to 1.7 eV. **Method:** SiC synthesis usually requires high energy but this study used Magnesiothermic Reduction, a low cost and low energy method in the synthesis of Silicon Carbide. Different magnesium loading was used to synthesized Silicon Carbide. **Results:** The synthesis was successfully done as supported by SEM-EDX and FTIR analyses. The measured band gap energies of the synthesized SiC from coconut shell regardless of the magnesium loading was about 2.1 eV. Doping the products using different concentrations of Na_2HPO_4 results in the lowering of the band gap energy to as low as 1.39 eV. **Conclusion:** Silicon carbide can be synthesized using magnesiothermic reduction method and doping the products promises a potential for photovoltaic solar cell application.

Keywords: Coconut Shell, Silicon Carbide, Magnesiothermic Reduction, Solar Photovoltaic Cell.

LITERATURE CITED

- Adams M., (2013). Silicon Carbide, SiC Ceramic Properties (Nov 2017) *Materials* retrieved from <http://accuratus.com/silicar.html>
- Abderrazak, H., & Hmida, E.S.B.H. (2011). Silicon Carbide: Synthesis and Properties. In *Properties and Applications of Silicon Carbide*. InTech.
- Akande, S. P., Oyedepo, O. J., & Olanitori, L. M. (2015). Performance of coconut shell ash and palm kernel ash as partial replacement for cement concrete. *Journal or Building Materials and Structures*, 2(1), 18-24.
- Angeles-Chávez, C., Espindola-Gonzalez, A., Martinez-Hernandez, A. L., Castano, V. M., & Velasco-Santos, C. (2010). Novel crystalline SiO₂ nanoparticles via annelids bioprocessing of agro-industrial wastes. *Nanoscale research letters*, 5(9), 1408.
- Arancibia-Bulnes, C.A, Tanori-Cordova J. C., Estrada, C. A., Cabanillas-Lopez, R. E. Ceballos-Mendivil, L. G., Perez-Rabago, C. A., Villafan-Vidales, H. I., & Murrieta-Yescas R. (2015). Synthesis of silicon carbide using concentrated solar energy. *Solar Energy*, 116, 238-246.
- Apasi, A., Madakson, P. B., & Yawas, D. S. (2012). Characterization of coconut shell ash for potential utilization in metal matrix composites for automotive applications. *International journal of engineering science and technology*, 4(3), 1190-1198.
- Baconguis, S. R. (2007, October). Abandoned biomass resource statistics in the Philippines. In *Proceedings of the 10th National Convention of Statistics, Manila, Philippines* (pp.1-2)
- Bai, Z. G., Dai, Z., Wang, Z., Gao, R. P., & Gole, J. L. (2000). Side-by-side silicon carbide-silica biaxial nanowires: Synthesis, structure, and mechanical properties. *Applied Physics Letters* 77, no. 21 (2000): 3349-3351
- Borchardt, L., Lee, J. T., Zhao, Y., Thie,e. S., Kim, H., Oschatz, M., ... & Yushin, G (2013). Sulfur-Infiltrated Micro- and Mesoporous Silicon Carbide-Derived Carbon Cathode for High-Performance Lithium Sulfur Batteries. *Advanced Materials*, 25(33), 4573-4579
- Borchardt, L., Korenblit, Y., Rose, M., Kockrick, E., Kvit, A., Kaskel, S., & Yushin, G. (2010). High-rate electrochemical capacitors based on ordered mesoporous silicon carbide-derived carbon. *American Chemical Society Nano*, 4(3), 1337-1344.

- Cabanillas-Lopez, R. E., Ceballos-Mendivil, L. G., Tanori-Cordova, J. C., Murrieta-Yescas, R., Zavala-Rivera, P., & Gonzales, J. C. (2014). Synthesis and characterization of silicon carbide in the application of high temperature solar surface receptors. *Energy Procedia*, 57, 533-540.
- Channiwala, S. A., Ghosal, G. K., & Parikh, J. (2007). A correlation for calculating elemental composition from proximate analysis of biomass materials. *Fuel*, 86(12), 1710-1719.
- Chen, P., Chen, D. H., Shi, Y. F., Meng, Y., Yang, H. F., ... & Zhao, D. Y. (2006). Highly ordered mesoporous silicon carbide ceramics with large surface areas and high stability. *Advanced Functional Materials*, 16(4), 561-567.
- Cheong, K. Y., & Chiew, Y. L., (2011). A review on the synthesis of SiC from plant-based biomasses. *Materials Science and Engineering: B*, 176(13), 951-964.
- Cho, K. M., Kim, K. H., Lee, D. J., Kim, S. J., Park, J. K., & Jung, H. T. (2015). Complete magnesiothermic reduction reaction of vertically aligned mesoporous silica channels to form pure silicon nanoparticles. *Scientific reports*, 5.
- Cho, E. C., Park, S., Hao, X., Song, D., Conibeer, G., Park, S. C., & Green, M. A. (2008). Silicon quantum dot/crystalline silicon solar cells. *Nanotechnology*, 19(24), 245201.
- Coconut Statistics (2015, October 29). Retrieved November 27, 2017, from <http://pca.da.gov.ph/index.php/2015-10-26-03-15-57/2015-10-26-03-22-41>.
- Costantini, J. M., Miro, S., & Pluchery, O. (2017). FTIR study of silicon carbide amorphization by heavy ion irradiations. *Journal of Physics D: Applied Physics*, 50(9), 095301.
- Dai, H., Wang, H., Li, X., & Robinson, J. T. (2009). Solvothermal reduction of chemically exfoliated graphene sheets. *Journal of the American Chemical Society*, 131(29), 9910-9911.
- Deng, D., Pan, X., Yu, L., Cui, Y., Jiang, Y., Qi, J., ... & Sun, G. (2011). Toward N-doped graphene via solvothermal synthesis. *Chemistry of Materials*, 23(5), 1188-1193.
- Dharma, J., Piscal A., & Shelton, C. T. (2009). Simple method of measuring the band gap energy value of TiO₂ in the powder form using a UV/Vis/NIR spectrometer. *Application Note Shelton, CT: PerkinElmer*.

- Dunlop, E. D., Green M. A., Emery, K., Hishikawa, Y., & Warta, W. (2015). Solar cell efficiency tables (Version 45). *Progress in photovoltaics: research and applications*, 23(1), 1-9.
- Ferreira, R., Sadi, S. H. F., Khanna, R., Rajarao, R., & Sahajwalla, V. (2014). Synthesis of silicon carbide nanoparticle by using electronic waste as a carbon source. *Materials Letters*, 120, (65-68).
- Gao, P. C., Lei, Y., Perez, A. F. C., Rajoua, K., Zitoun, D., & Favier, F. (2011). New topotactic synthetic route to mesoporous silicon carbide. *Journal of Materials Chemistry*, 21(39), 15798-15805.
- Gerbec, J. A., Lin, H., Sushchikh, M., & McFarland, E. W. (2008). Synthesis of amorphous silicon carbide nanoparticles in a low temperature low pressure plasma reactor. *Nanotechnology*, 19(32), 325601.
- Gersten, B. Solvothermal Synthesis of Nanoparticles (n.d.) Retrived December, 2017, from <https://www.sigmaaldrich.com/technical-documents/articles/chemfiles/solvothermal-synthesis.html>
- Gopal, V. V., & Kamila, S. (2017). Effect of temperature on the morphology of ZnO nanoparticles: a comparative study. *Applied Nanoscience*, 7(3-4), 75-82.
- Gregorio G. C. Agricultural Waste in the Philippines (2017). Retrieved November 2017, from <https://jcgregsolutions.wordpress.com/2017/02/27/agricultural-waste-in-the-philippines.html>
- Greyson, E. C., Barton, J. E., & Odom, T. W. (2006). Tetrahedral Zinc Blende Tin Sulfide Nano-and Microcrystals. *Small*, 2(3), 368-371.
- Guo, X. Y., & Jin, G. Q. (2003). Synthesis and characterization of mesoporous silicon carbide. *Microporous and mesoporous materials*, 60(1), 207-212.
- Guo, X. Y., Jin G. Q., & Liang, P. (2003). Novel method for synthesis of silicon carbide nanowires. *Journal of materials science letters*, 22(10), 767-770.
- Han R., Xu X., Hu X., Yu N., Wang J. Tan Y. Huang W., (2003), Development of bulk SiC single-crystal grown by physical vapor transport method, *Optical materials*, 23, pp: 415-420
- Heidarzeh, H., Rostami, A., Dolatyari, M., & Rostami, G. (2014) Effect of Dopant Concentrations on Conversion Efficiency of SiC-Based Intermediate Band Solar Cells, *International Congress on Energy Efficiency and Energy Related Materials*, 15, 120-124.

- Kalbasi, R. J., Saeedifar, Z., Nourbakhsh, A., & Karamian, E. (2013). Low-temperature magnesiothermic synthesis of mesoporous silicon carbide from an MCM-48/polyacrylamide nanocomposite precursor. *Journal of Materials Science & Technology*, 29(3), 255-260.
- Kumar, L., Pandey, K. K., & Khan, S. (2017). Use of Coconut Shell Ash as Aggregates.
- Launer, P., & Arkles B., (2013). Infrared Analysis of Organosilicon Compounds: Spectra-Structure Correlations. *Silicon Compounds: Silanes & Silicones*, 175-178
- Moissanite vs Diamond (Nov 2017) retrieved from <https://www.brilliantearth.com/news/moissanite-vs-diamond/>
- Mokaya, R., Yang, Z., & Xia, Y., (2004). High surface area silicon carbide whiskers and nanotubes nanocast using mesoporous silica. *Chemistry of Materials*, 16(20), 3877-3884.
- Nair, N. G., Selvam, A., & Singh P. (1998). Synthesis and characterization of SiC whiskers from coconut shells. *Journal of materials science letters*, 17(1), 57-60.
- Niu, Y., Zhang, X., Zhao, J., Tian, Y., Li, Y., & Yan, X. (2014). Preparation, characterization and properties of amine-functionalized silicon carbide/polyimide composite films. *Royal Society of Chemistry Advances*, 4(54), 28456-28462.
- Production quantities of coconuts by country (Nov 2017) FAOSTAT, retrieved from <http://www.fao.org/faostat/en/#data/QC/visualize>.
- Patil, R., Dongre, R., & Meshram, J. (2014). Preparation of silica powder from rice husk. *Journal of Applied Chemistry*, 27, 26-29.
- Raveendran, K., Ganesh, A., & Khilar, K. C. (1996). Pyrolysis characteristics of biomass and biomass components. *Fuel*, 75(8), 987-998.
- Rajarao, R., & Sahajwalla, V. (2016). A cleaner, sustainable approach for synthesizing high purity silicon carbide and silicon nitride nanopowders using macadamia shell waste. *Journal of Cleaner Production*, 133, 1277-1282.

- Shu, W., Liu, Y., Peng, Z., Chen, K., Zhang, C., & Chen, W. (2013). Synthesis and photovoltaic performance of reduced graphene oxide–TiO₂ nanoparticles composites by solvothermal method. *Journal of Alloys and Compounds*, 563, 229-233.
- Silicon Carbide (Nov 2017), *Silicon Carbide (SiC) Nanoparticles, Properties and applications* retrieved from <https://www.azonano.com/article.aspx?ArticleID=3396>
- Silicon Carbide (laboratory grown) from Ontario Canada. Online Mineral Museum, 2017
- Sivasubramanian, S., & Sravanthi L. (2015). Synthesis and characterization of silica nano particles from coconut shell. *International Journal of Pharma and Bio Science*, 6(1), 530-536.
- Solar Central (2017, December). *P/N Junctions & Band Gaps* retrieved from http://solarcellcentral.com/junction_page.html
- Solar Cells. (2017, December). Retrieved from <http://www.explainthatstuff.com/solarcells.html>
- Su, J., Chen, Z., Gao, B., Fu, J., An, W., Peng, X., ... & Chu, P. K. (2016). Large-scale synthesis and mechanism of β -SiC nanoparticles from rice husks by low-temperature magnesiothermic reduction.
- Table of IR Absorptions (2018, May 22). Retrieved from <https://webspectra.chem.ucla.edu/irtable.html>
- Takahiro, M., Yoshitake, K., Taku, Y., Naoki, S., & Jun, A. (2008). Superconductivity in carrier-doped silicon carbide. *Science and Technology of Advance Material*, 9, 1-8.
- Turangan, T. M., Tiwow, V. A., & Simandjuntak, S. (2017). Of Coconut Shellwaste Pyrolysis Potent as a Carbon Material From Minahasa Charcoal Cy, Indonesia. *International Journal of Applied Chemistry*, 13(3), 721-731.
- Trivedi, M. K., Nayak, G., Tallapragada, R. M., Patil, S., Latiyal, O., & Jana, S. (2015). Effect of biofield treatment on structural and morphological properties of silicon carbide.
- Wesch, W. (1996). Silicon carbide: synthesis and processing. *Nuclear Instruments and Methods in Physics Research Section B: Beam interactions with materials and atoms*, 116(1-4), 305-321.

- Wu, X. L., Fan, J. Y., Qiu, T., Yang, X., Siu, G. G., & Chu, P. K. (2005). Experimental evidence for the quantum confinement effect in 3 C-SiC nanocrystallites. *Physical review letters*, 94(2), 026102.
- Zhao, B., Zhang, H., Tao, H., Tan, Z., Jiao, Z., & Wu, M. (2011). Low temperature synthesis of mesoporous silicon carbide via magnesiothermic reduction. *Materials Letters*, 65(11), 1552-1555.